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MODIFICATION OF A SCANNING ELECTRON MICROSCOPE FOR REMOTE OPERATION IN A HOT CELL

Introduction

The scanning electron microscope (SEM) has become an invaluable tool for the characterization of the failure mode in fracture and fatigue studies because of its depth of field and wide range of magnification. To adequately conduct fracture and fatigue studies on irradiated materials it is essential to have a shielded facility where highly radioactive specimens can be handled remotely during loading and examination in the SEM. Only three such facilities have been described in the literature (1-3). This report describes the modifications required to adapt an International Scientific Instruments (I.S.I.) Super II SEM for remote operation in a shielded facility and describes the facility at NRL and its' operation.

Analysis of Problem

The specimens to be examined in the SEM consisted of fracture surfaces of Charpy V-notch specimens, compact tension fatigue and fracture toughness specimens in sizes ranging from $1/2 T$ to $2 T$, and single edge notch fatigue bend bars of 1.0 in. thickness. These broken fracture specimens of unirradiated materials are normally sectioned approximately $1/4$ inch below the fracture surface by sawing or abrasive cut-off wheel to preserve the entire fracture surface. Care must be exercised not to damage the surface in any way, either mechanically or by corrosion. Materials to be examined included pressure vessel steels, 12% Cr martensitic stainless steels, austenitic stainless steels, nickel base alloys and titanium alloys. Some of the specimens to be examined had been irradiated to neutron fluences in the high 10^{22} n/cm² range and thus were highly radioactive.

It is obviously desirable to reduce the mass of radioactive material to be handled to a minimum but a loss of information will result from any damage

to the fracture surface resulting from attempts to make too thin a section or from sections through the surface. The above considerations indicated that sources of γ radiation of the order of 400 millicuries with radiation levels above 0.5 Roentgen/hr at 1 meter would be encountered.

The normal sequence in loading an unirradiated specimen into the SEM involves mounting the specimen onto a stub by pressing onto a strip of adhesive conductive tape and then locking the stub in place with an Allen head screw. A modification of the standard operating procedures was clearly required to examine radioactive specimens.

The large source strengths to be handled dictated that a hot cell would be required as opposed to a shielded facility. Manipulators suitable for remote loading of the radioactive specimens would also be required. The requirement that the SEM be placed in a hot cell strongly favored designs where the electron optical column (and attached specimen chamber) was a separate component from the control console. The unit selected was the I.S.I. Super II SEM.

The I.S.I. Super II SEM is a compact SEM possessing the basic capabilities to form an image of the fracture surface with secondary or back-scattered electrons. An electron beam emitted from an electron gun is accelerated by a high voltage, focused by a three-stage electro-magnetic lens system and scanned over the specimen surface. The secondary electrons are collected by a detector, converted to video signals, displayed on a Cathode Ray Tube (CRT) and magnified electronically in synchronization with the scanning beam in the electron column. The display of the specimen surface on the CRT is recorded on Polaroid film. The electron-optical column and specimen chamber are an integral unit. The control console, rotary vacuum pump, and high voltage generator are connected to the optical column using hoses and electrical cables.

The specimen loading is accomplished by opening the chamber door, loosening the holder cup locking screw, placing a specimen holder (on which a specimen has been previously mounted) on the stage, and retightening the set screw. The chamber door is then closed and locked. The specimen is positioned with hand operated X and Y controls located on the specimen chamber. Likewise, the Z control, rotation, tilt, and specimen stage clamp controls are hand operated.

The microscope as supplied by the manufacturer required a number of modifications to adapt it for operation in the hot cell. Extended cables from the electron optical column to the power supply and from the detector to the control console were available from the manufacturer. Other modifications to permit alignment using tools operated with the manipulators, remote drives for the stage controls, and remote specimen loading were designed and developed at NRL. These modifications and additions to the microscope were required so that all necessary adjustments and specimen manipulations could be made from the operator's station at the cell face and are described in the following section.

Modifications of I.S.I. Super II SEM For Operation in Hot Cell

The optical column of the SEM was placed inside a small hot cell which had a lead glass viewing window and portholes for physical access. The SEM control console, high voltage generator, vacuum rotary pump, and associated equipment were located at the front face of the cell. Hoses and electrical cables which penetrated the cell wall were used to interconnect the optical column and the control equipment located at the cell face. The various major components including modifications of the system are shown in Fig. 1.

One modification required was the relocation of the three-position vacuum control selector switch from the optical column to the remote control unit so as to permit the operator to control the vacuum system valving operations from the cell face location. See Fig. 1 for the new location. A second modification involved the X and Y electron gun mechanical alignment controls which center the electron beam with respect to the column. In the normal mode of operation the gun is translated in the plus X direction by tightening thumb screw X_1 and at the same time loosening thumb screw X_2 as shown in Fig. 2a. The minus X translation was done by loosening X_1 and tightening X_2 . The plus and minus Y electron gun translations were done in the same manner using Y_1 and Y_2 thumb screws. This method of alignment was awkward and not compatible with the constraints of hot cell operations. A new and simplified alignment mechanism was designed to eliminate the thumb screw centering procedure. The mechanism which is illustrated in Fig. 2b and seen in Fig. 1, operates as follows: The operator rotates the X direction "T" handle screw clockwise with the manipulator, to translate the electron

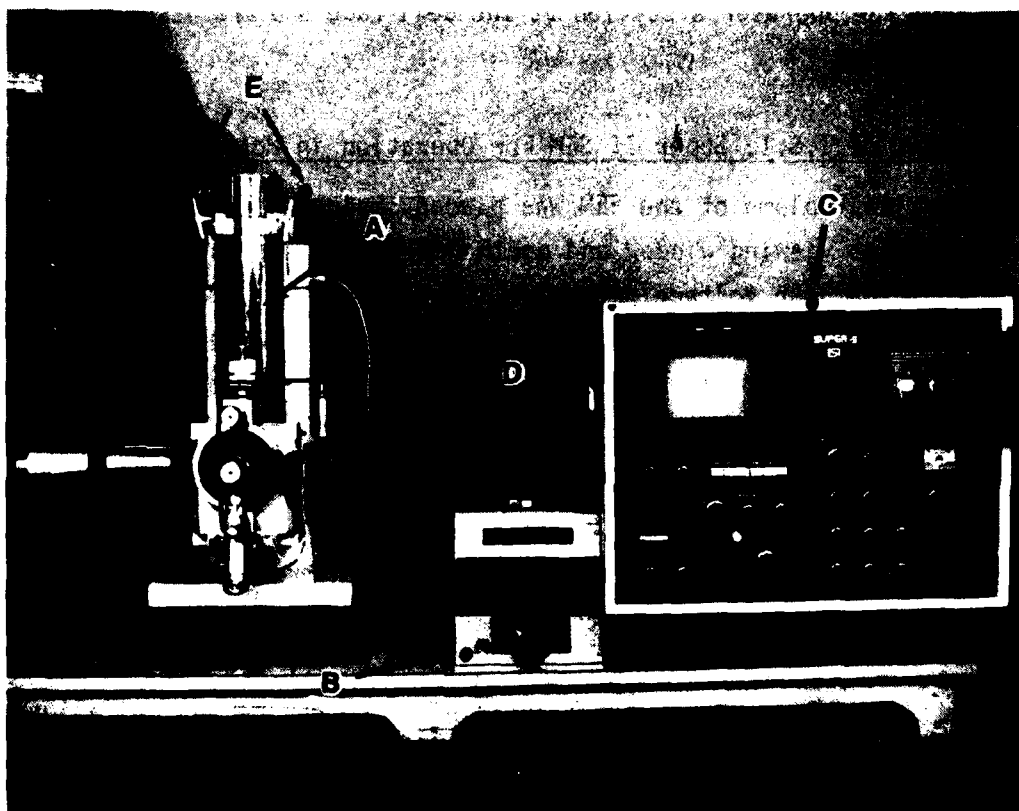


Fig. 1. A view of the Scanning Electron Microscope system including modifications necessary for hot cell installation. A is the optical column, B the specimen stage remote control unit, C is the SEM control console, D is the three-position vacuum control selector switch, and E the mechanical electron gun alignment controls.

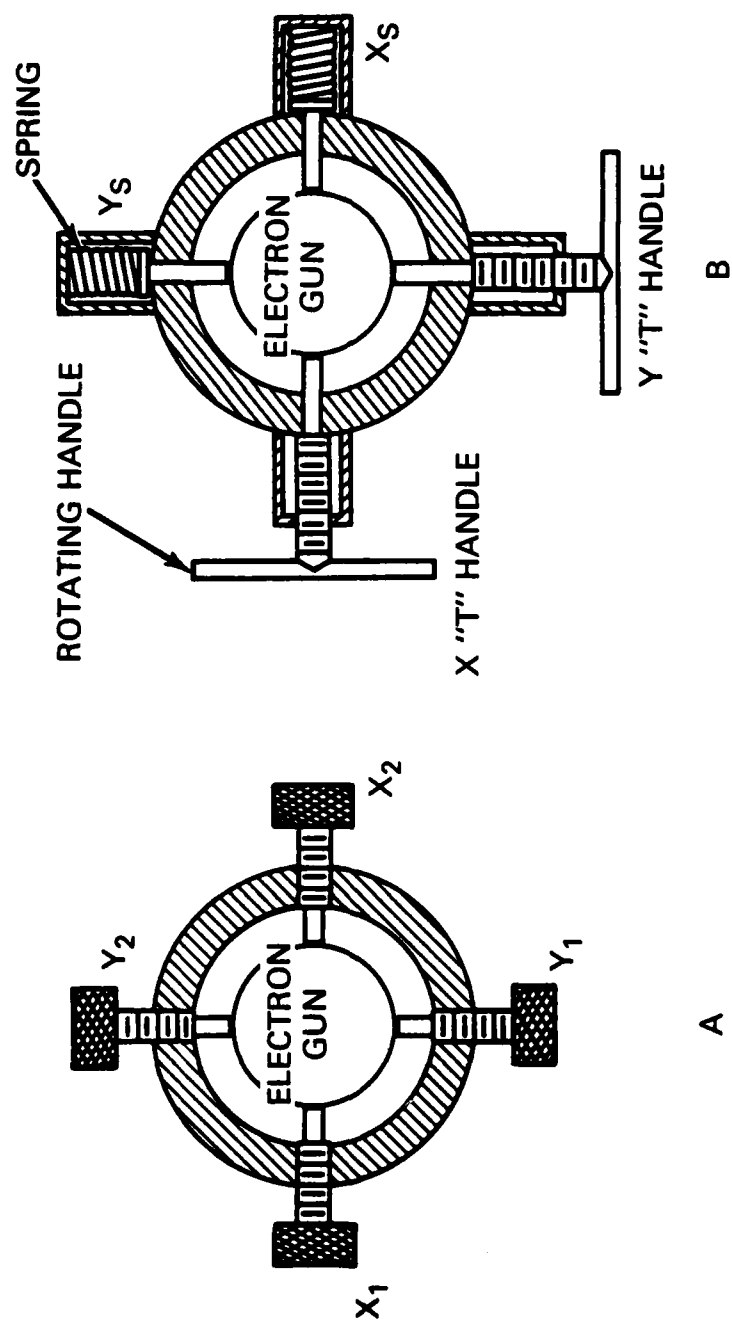


Fig. 2. (A & B) Two mechanical electron gun alignment mechanisms for a SEM.
A illustrates a thumb screw drive system and B a "T" handle screw drive and spring return system.

gun against spring X_s . The spring exerts an opposing force sufficient to insure positive position control and also reverse translation of the gun when the screw is rotated counter clockwise. Thus, both plus and minus X electron gun translation are possible by rotating one adjustment screw from the cell face location. The Y translation is achieved in an identical fashion using a second "T" handle.

Filaments for the electron gun commonly have a lifetime of about 40 hr so it was desirable to have the capability of changing filaments remotely with the manipulators. After some experimentation a procedure was developed in which the filaments were placed in the cap assembly by hand and passed into the hot cell through a cell face loading port. The pre-loaded filament cap assembly was then inserted into the electron gun with the manipulators. Alignment of the electron gun was then accomplished as described in the previous paragraph.

Extensive modifications were required for remote loading of the specimen and for the stage motion controls. A new specimen holder shown in Fig. 3 was designed to meet the hot cell requirements. The two part holder uses a spring loaded ball (Part 1) to insure that the specimen mounting stem (Part 2) is clamped tightly to provide mechanical stability and electrical continuity. Part 1 is permanently attached to the specimen stage by the holder cup locking screws. The taper design insures that the two parts will not lock up and also makes it easier for the operator to remotely load and unload the specimens. The sample is attached to Part 2 with electro-conductive adhesive foil. A large supply of interchangeable Part 2 pieces provides flexibility for sample loading operations.

Stage controls for the Super II allow translation of the specimen in the X, Y, and Z directions for specimens observation, motion in the Z direction for focusing, tilt of the stage for optimizing the electron collection conditions and for stereo pairs, and rotation of the specimen for proper positioning. All these controls are used during examination of a specimen and the X and Y translate controls especially are used almost continuously when scanning the specimen for significant features. Normally, all the above controls are manually operated. The fine degree of control and adjustment required to position the specimen for viewing at high magnifications precluded the use of the manipulators to operate the controls. A system using

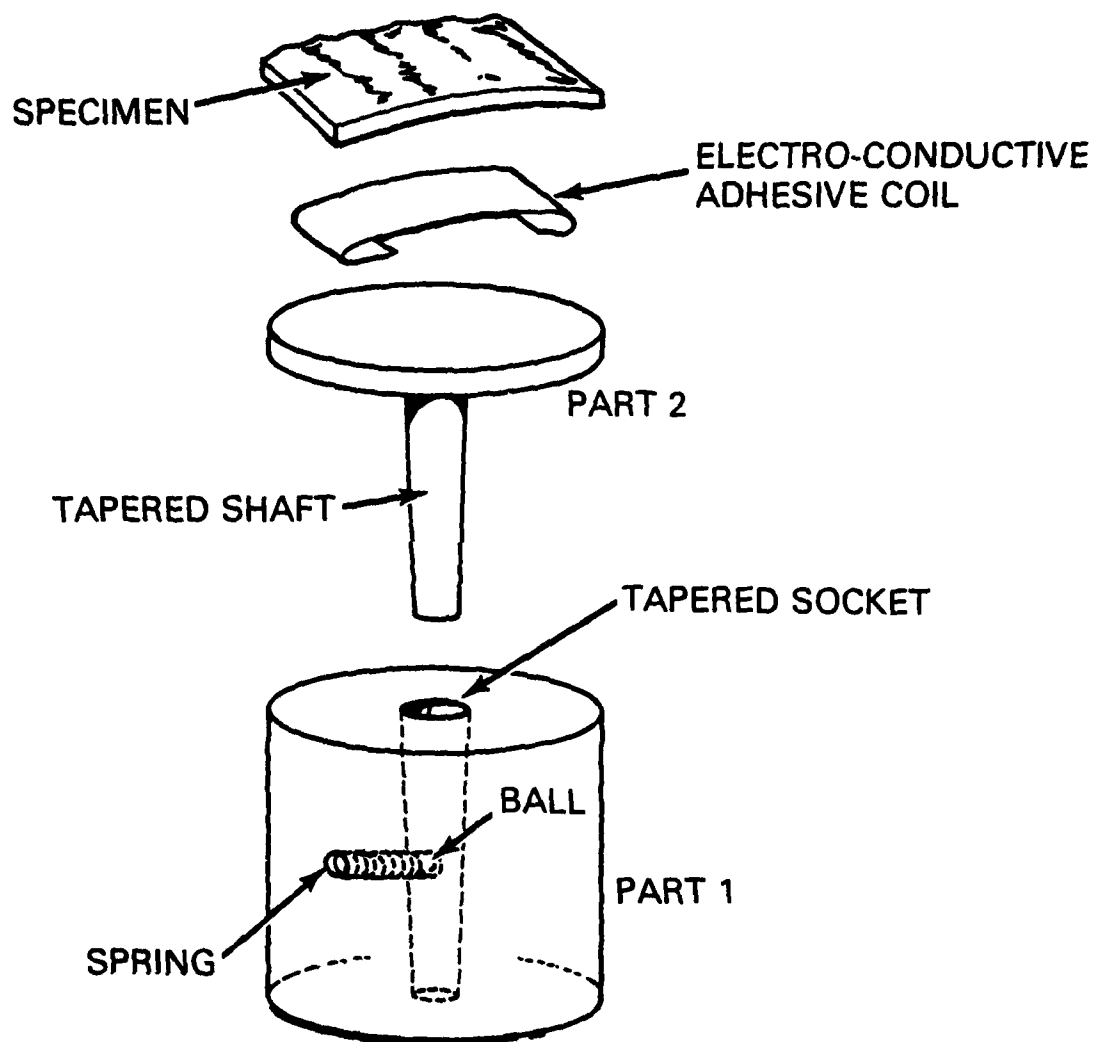


Fig. 3. A two-part Scanning Electron Microscope specimen holder designed to simplify remote loading of radioactive specimens

four precision drive motors and "O" ring drive belts was developed so that the stage functions could be operated and controlled from outside the hot cell. The drive system and mounting hardware are shown in Fig. 4. A 12 volt D.C. power supply provides power to the four D.C. permanent magnet planetary gear motors supplied by Globe Manufacturing Co. The speed and direction of each motor is controlled from the SEM Stage Remote Control unit shown in Fig. 1 (Component B). A function selector switch allows selection of either X, Y, or Z translation or rotation of the specimen. A fast-slow switch selects the desired speed, and a direction switch selects the direction of translation or rotation. Only one movement at a time is permitted. A foot switch is used to start and stop the selected drive motor. The stage tilt control is used less frequently than the X, Y, Z and rotation controls and requires less precision in positioning. It was concluded that tilt motion of the stage could be adequately controlled with the manipulators by exerting a rotational force on the main clamp ring. It was necessary to fabricate a new tilt position indicator that could be read through the hot cell window. The modification is shown in Fig. 4.

Hot Cell Facility

The existing radiation facility at NRL consisted of five hot cells which were dedicated as testing and machine shop cells and thus were not suitable for mutual use because of the high level of contamination and space-sharing conflicts. Because of the desirability to control contamination and limit personnel exposure to radiation, a new hot cell dedicated to the SEM was constructed.

The High Level Radiation Laboratory (HLRL) hot cell facility as originally designed and constructed contained five continuous cells with seven work stations. Space was made available at each end of the row of hot cells to add an additional cell. However, it was not practical from a cost standpoint to construct a new dedicated cell for the SEM. To meet the needs of the SEM, a surplus, free standing hot cell was obtained. The cell was modified and installed so that the front face is flush with a wall which adjoins the cell and separates the "cold" work area from the "warm" work area (Fig. 5).

The cell consists of a shielded area, four walls and a top which is

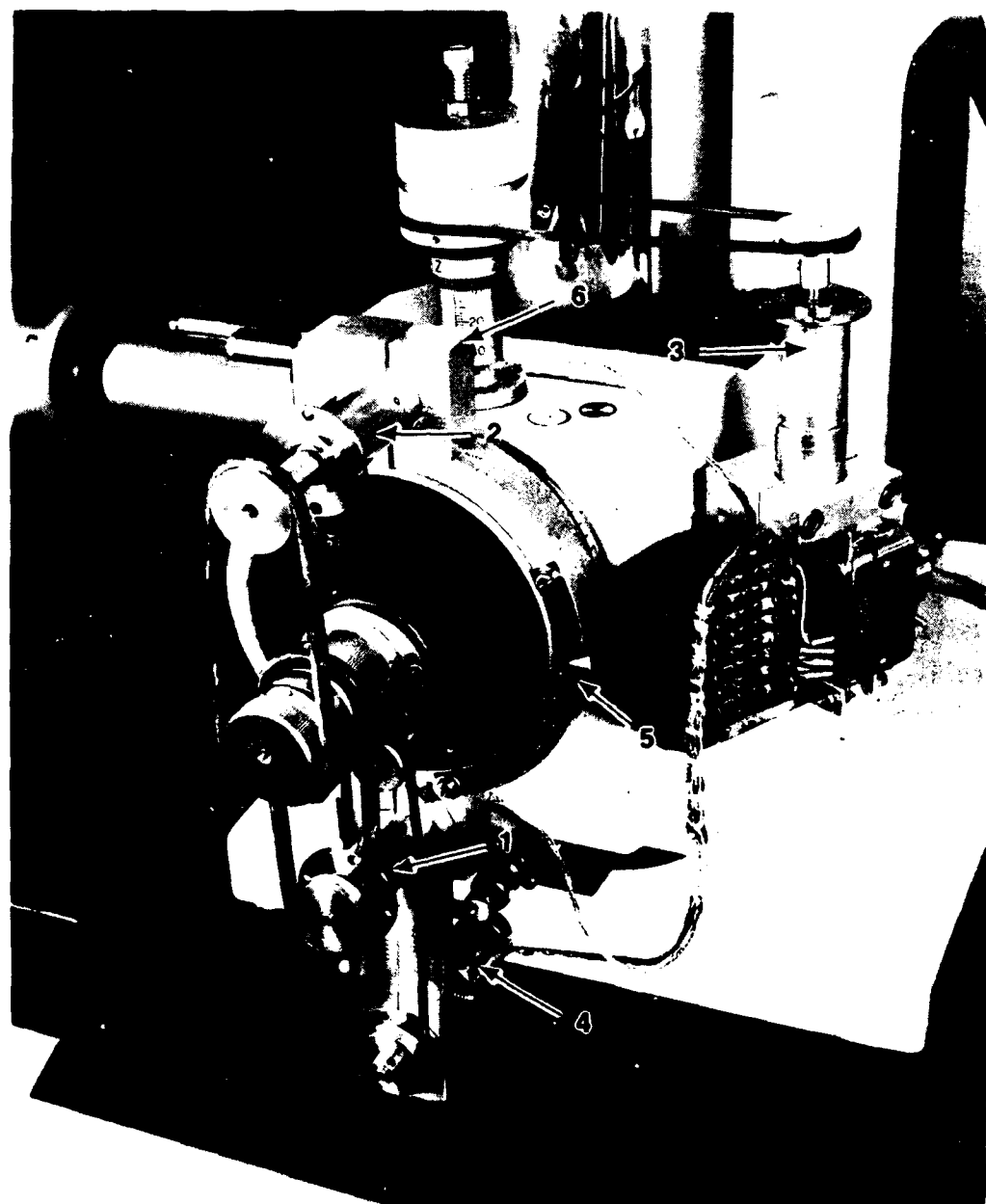


Fig. 4. Specimen stage modifications of the Scanning Electron Microscope for hot cell installation: (1) X translation drive motor; (2) Y translation drive motor; (3) X translation drive motor; (4) Rotation drive motor; (5) stage tilt indicator; and (6) Main clamp ring.

split into two pieces for ease in installation. The base, front and side walls consist of 6 in. of lead; the top is 4 in. of lead. The rear wall is composed of steel plates which are split so that the bottom section can be removed for easy access to the SEM. Additional plates can be added if shielding requirements dictate. The inside measurements are 5 ft. in all three directions.

Two Central Research Model G master slave manipulators are installed in the front wall. The front wall also has three ports which may be used for equipment or unirradiated specimen pass-through (Fig. 5). Additional ports are provided on the side walls and ample space is available for the large volume of cables connecting the microscope to the control console.

A remotely controlled elevator was fabricated and installed below an opening in the cell floor which was formerly used as the hot cell exhaust port. Samples are transferred to the elevator in a small transfer cask then raised up into the cell where the specimen can be safely removed.

The cell was originally supplied with its own absolute filtering and exhaust system. This was abandoned in favor of utilizing the existing HLRL hot cell exhaust and filtering system. A port located near the top of the wall facing the existing hot cells was connected by a duct to the cell exhaust system and provided with a damper to control the amount of air flow.

The cell can be easily modified to meet new requirements by utilizing the availability of shielded ports and the flexibility provided by the rear wall.

Operating Experience

At the time of this writing the SEM hot cell facility has been in service for routine operations for 26 months and 40 radioactive specimens have been examined. Experience for the more common specimen handling and microscope service operations are described below to demonstrate the utility of the design. An overall view of the SEM hot cell facility may be seen in Fig. 5.

One of the major initial concerns was the degradation of the scintillator lifetime as a result of exposure to high level gamma radiation. After a total of 30 months of operation (including 4 months of instrument check-out

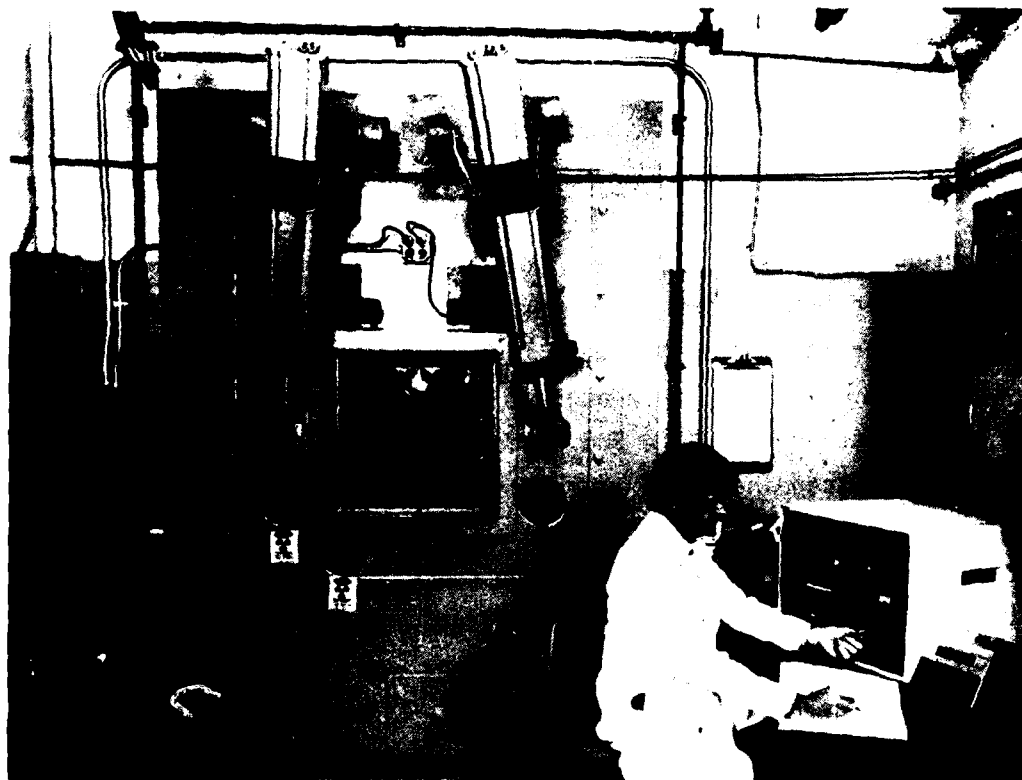


Fig. 5. A view of the Scanning Electron Microscope hot cell facility for study of highly radioactive materials.

time), the scintillator crystal has received an estimated exposure of 500 R. The maximum exposure levels at the scintillator were ≈ 4 R. Some deterioration in image quality was observed and is attributed to normal usage and shelf life of the scintillator. Gamma radiation at these levels apparently does not decrease the life of the scintillator noticeably. Scintillator lifetimes in the practical range are therefore possible.

Another major concern was the effectiveness of the radiological shielding provided by the hot cell. The radiation level from one radioactive specimen which read 500 mR/hr at 1 m (estimated source strength of 400 mCi) was attenuated to an exposure rate of 0.5 mR/hr at the operator's position (also 1 m away) by the column shielding and the hot cell wall.

Time and motion studies were conducted for several typical specimen handling and maintenance activities. A specimen which had been mounted on the specimen holder (part 2 in Fig. 3) with conductive adhesive tape in an operation outside the hot cell was loaded onto the specimen stage using the manipulators in two minutes. Specimen scanning with the mechanized drive was actually faster for the higher magnifications (50,000 to 100,000 X) because the vibration level was low enough to permit continuous scanning. At the lower magnifications (30 X to 500 X) however, a higher speed drive would be desirable when scanning areas in excess of 2 cm². Filament replacement was required during one specimen examination and was accomplished remotely from the cell face in about ten minutes. The radioactive specimen was not removed or disturbed during the filament exchange. Mechanical alignment of the electron gun was required after the filament was replaced and this was also done remotely using the manipulators and the "T" handle tool to re-center the electron gun. The alignment procedure required about five minutes.

During normal operations the column liner becomes contaminated by hydrocarbons from the pump oil and degassing of the specimen. The hydrocarbons deposit on the column liner and build up an electrical charge which produces distortion in the electron beam. The column liner must therefore be cleaned periodically (typically once a month for an 80% use factor). This operation requires access to the column so the back of the hot cell must be removed, the column liner removed and cleaned and then reassembled and reinstalled in the column. The column liner and stage were monitored for radioactive contamination at the time of disassembly during one periodic clean-

ing. No contamination was found on any part of the column assembly but the stage was reading 15 mR/hr at contact. This level was reduced to 2 mR/hr at contact by vacuum cleaning to remove the loose contamination. The entire operation of hot cell opening, column liner cleaning and replacement, decontamination, reassembly and hot cell closure required six hours.

Another maintenance case involved failure of the vacuum selector valve located on the optical column. Air was inadvertently admitted to the hot diffusion pump and the oil cracked. The vacuum selector valve and diffusion pump were disassembled and checked for radioactive contamination and were found to be free of contamination on the interior surfaces. The exterior of each was slightly contaminated and required normal precautions for handling. The vacuum valve required replacement of three new "O" rings and the diffusion pumps required a thorough cleaning and the addition of new oil to return the system to operational condition. This entire procedure, which required removing the back of the hot cell, was completed in one and one-half working days.

User satisfaction with the facility has been quite high. Image quality and stability appear to be comparable to out-of-cell conditions. The major concern in this regard is vibration isolation and no modifications beyond the standard vibration isolation pad supplied with the unit have been required. As previously noted, low magnification scanning operations are somewhat slow and a higher speed drive motor would be desirable. Tilt operations for stereo pairs have also been found to be somewhat cumbersome and accuracy of positioning is limited to ± 0.5 deg with the present manipulator mode of positioning the tilt control. In general, the system has proven to be highly successful and has satisfied the initial design objectives for a low cost remotely operated SEM for examination of highly radioactive specimens.

Conclusions

Highly irradiated fracture and fatigue specimens can be easily examined in a new SEM hot cell facility at NRL. Image quality is not degraded by the high level γ irradiation fields, the necessary specimen preparation, manipulation and examination procedures can be carried out in the hot cell, and routine SEM operations and maintenance can be accomplished remotely in reasonable times.

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